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## THE EFFECT OF CELLULOSE ACETATE FIBER AND FILTER PARAMETERS ON INTERMEDIATE ROD FIRMNESS

### Introduction

As part of ongoing support of the cigarette industry, Celanese presented the following paper at the 37th Tobacco Chemists' Research Conference in Arlington, Virginia, USA.

The effects of filter specifications on fully-cured rod firmness, or firmness after at least twenty-four hours of cure time, have been well documented. Intermediate firmness, on the other hand, has received limited attention but is of great significance to the cigarette manufacturer. Intermediate firmness is defined in this study as the filter rod rigidity between fifteen minutes and two hours after manufacture. Cigarette filter rods must meet a certain minimum firmness to withstand the filter rod and cigarette making processes. Soft rods will result in unacceptable filter tip appearance and increased reject levels of cigarettes.

The present study focused on the effects of filter tow denier per filament and total denier, fiber effective crimp index, filter plasticizer level, and filter rod circumference on intermediate rod firmness.

Filter tow items were evaluated in which the denier per filament ranged from 2.0 - 6.5 and the total denier from 30,000 - 45,000. The filter rod circumference ranged from 22.2 - 25.8 millimeters and the plasticizer application level from 0 - 10 percent.

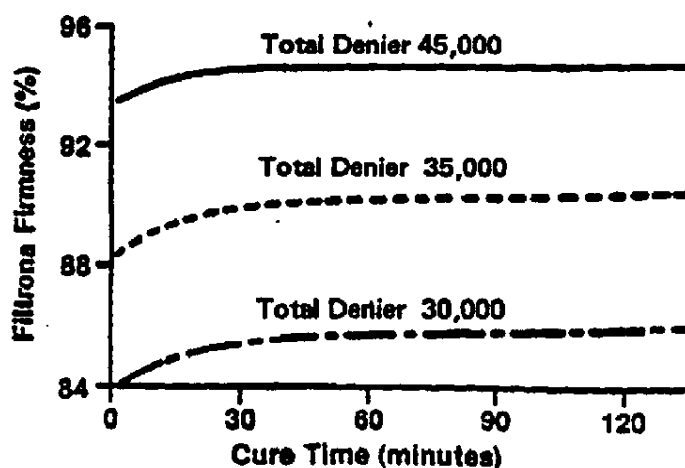
The fiber effective crimp index (ECI) was varied from 1.27 to 1.63. ECI is defined as the calculated weight of tow in the filter rod in grams/9000 meters divided by the uncrimped total denier of the cigarette tow used to manufacture the filter.

Results of this study indicated when all other filter specifications were held constant, intermediate firmness increased with increasing denier per filament. This may be attributed to the increasing rigidity of the filaments in the filter rod as denier per filament is increased.

Intermediate firmness also increased with increasing total denier resulting from the increasing fiber volume fraction in the filter rod (Figure 1).

Figure 1

### EFFECT OF TOTAL DENIER ON INTERMEDIATE FIRMNESS



# MENTHOL DISTRIBUTION AND TRANSFER

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Even after 7 weeks aging, menthol cigarettes in which all the menthol is initially applied to the filter do not have the same menthol distribution as those in which all the menthol is initially applied to the tobacco. In the former case, a stable cigarette is obtained after 4 weeks with 40% of the menthol being on the tobacco. In the latter case, a stable cigarette is obtained after 4 weeks in which 70% of the menthol remains on the tobacco. Although both cigarettes give similar total menthol deliveries to mainstream smoke (16% of the total menthol is transferred), the puff by puff menthol deliveries differ. Menthol deliveries increase fairly uniformly with puff number when 70% of the menthol is on the tobacco. Menthol delivery is extremely high in the last puffs when 60% of the menthol is on the filter. Aerosol size distribution, and the concentration of menthol in the TPM deposited on the filters, are considered to play a role in menthol elution from filters.

## INTRODUCTION

Curran (1) has described the migration of menthol from tobacco to cellulose acetate, and from cellulose acetate to tobacco. He found that for cigarettes aged 28 days, 80% of the menthol remained on the tobacco when all the menthol was initially on the tobacco, and that 60% of the menthol remained on the filter when all the menthol was initially on the filter. He anticipated that equilibrium would eventually be reached with 70% of the menthol on the tobacco regardless of the initial distribution. He also showed that per puff deliveries of menthol depended on the menthol distribution in the cigarette.

We present, in this paper, aging studies over a longer time period, the effect of menthol distribution on menthol transfer to mainstream smoke, and some suggestions on the mechanisms involved in the elution of menthol from filters.

## MATERIALS AND METHODS

### Cigarettes

The cigarettes, made from a typical blend of U.S. tobaccos, were 84 mm in length and 25 mm in circumference. The 21 mm filters were made from 8.8/44,000 Y cross section cellulose acetate tow. Menthol was applied to either the filter tow or to the tobacco prior to final cigarette assembly. In both cases, the loading was 2.7 mg on the finished cigarette. Menthol was applied to the filter in a triethylene glycol diacetate/polyethylene glycol plasticizer, Cigarette F; to the tobacco, from ethanolic solution, Cigarette T.

## ANALYSIS

### Menthol on Cigarette by Colorimetry

The menthol was removed from tobacco and/or filters by steam distillation. An aliquot was taken from the distillate, and a color developed with N,N-dimethyl-p-aminobenzaldehyde in the presence of 80% H<sub>2</sub>SO<sub>4</sub>(4). The absorbance was measured at 540 mμ against a reagent blank.

### Menthol in Smoke by GLC

Cigarettes were smoked according to standard FTC Contribution received: May 8, 1972. Tob. Sci. XV11; 10-11; 1973.

procedures (35 ml puff, two second duration, one per minute) using a Phipps and Bird smoking machine.

The Cambridge pads containing the smoke condensate were extracted with 20 ml of isopropanol with butylene glycol internal standard. Gas chromatograph was on a 3 ft x 1/8 in. stainless steel column packed with 80-100 mesh Chromosorb W (acid washed and silanized), programmed from 85° to 250°C. at 18.75°/min. The injector block was held at 220°C. and the detectors at 200°C. Helium flow at the detector outlet (4-1 splitter) was 25 ml/min.

## RESULTS AND DISCUSSION

### A. Menthol Distribution

Menthol distribution between tobacco and filter material over a 7 week aging period is shown in Figure 1 for each of the two cigarettes. A stable situation is reached after 28 days. When menthol is initially on the tobacco, the final distribution is 70% menthol on the tobacco. This value corresponds with that found by Curran (1), and is the distribution we have found for a number of commercial cigarettes, Table 1. When menthol is initially on the filter, only 40% migrates to the tobacco even after a 7 week aging period. There is, in fact, very little change in the distribution after 3 weeks. It would appear that, when the menthol is initially applied to the filter, part of it is irreversibly bound in with the filter material or plasticizer.

### B. Menthol Transfer to Smoke

Menthol transfer to mainstream smoke is shown in Figure 2 in terms of the distribution between the tobacco and the filter. Highest menthol transfer, about 80%, is found when most of the menthol is on the filter. High menthol transfers, around 20%, are also found when most of the menthol is on the tobacco. In

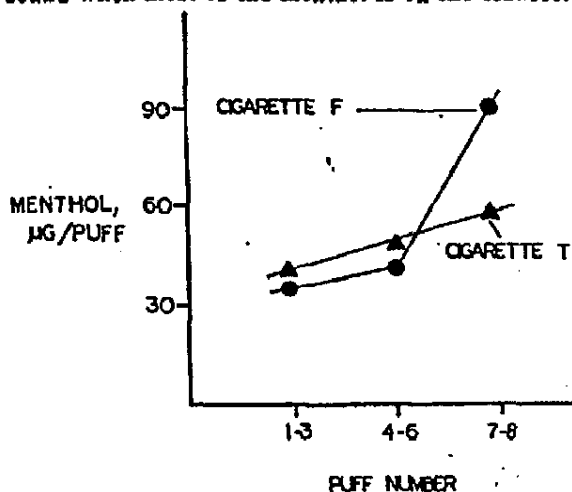


Figure 1. The distribution of menthol between tobacco and filter.

(Tobacco Science 10)

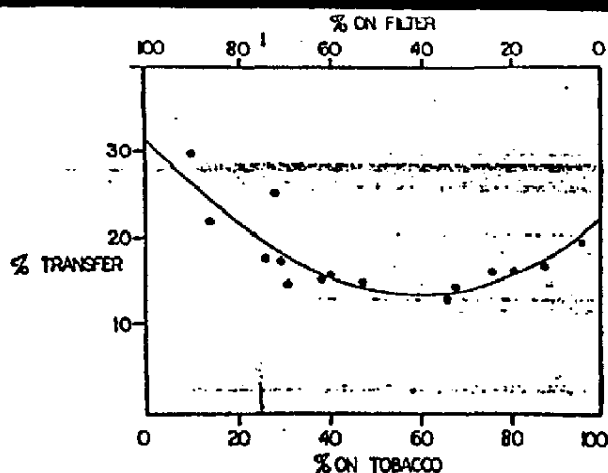


Figure 2. Menthol transfer to smoke from different menthol distributions.

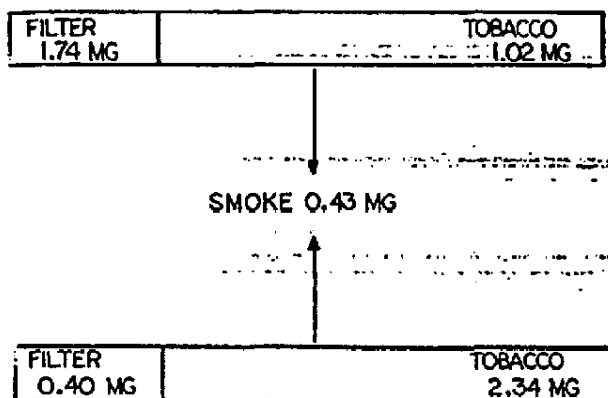


Figure 3. Equivalent menthol deliveries from different menthol distributions.

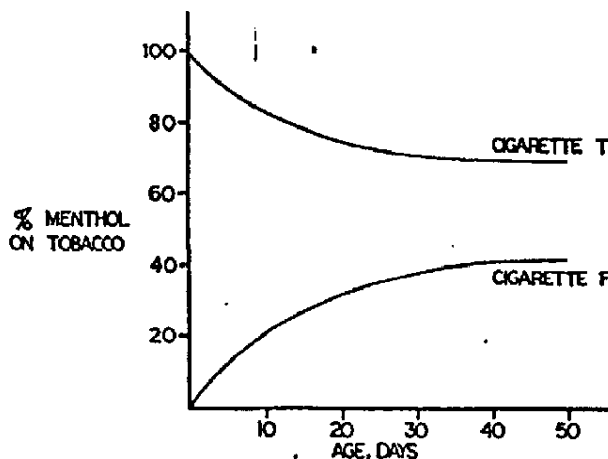


Figure 4. Per puff menthol deliveries from different menthol distributions.

the two stable regimes, 70% menthol on the tobacco, or 60% menthol on the filter, menthol transfers are about 16%. Thus, equivalent menthol deliveries are found from two cigarettes in which the menthol distribution is quite different, Figure 3.

The deliveries from these two cigarettes were examined in more detail. Menthol deliveries from puffs 1-3, 4-6, and 7-8 were determined after an aging period of

Table 1. Menthol distribution in commercial cigarettes

Brand	% Menthol on filter
A	33
B	27
C	35
D	30
E	29
F	27

Table 2. Factors affecting menthol elution

Factor	Approximate magnitudes
Filter Temperature	Early puffs 25°C, Last puffs 70°C
Aerosol Concentration	20 µg/ml
Aerosol Surface Area (S)	40 µg/ml
Menthol in TPM on Filter	20 mg/g
Cigarette T	4%
Cigarette F	16%

Table 3. Ratio of menthol to nicotine and puff number

Puffs	Cigarette T	Cigarette F
1-3	$1.11 \times 10^{-2}$	$0.82 \times 10^{-2}$
4-6	1.52	2.68
7-8	0.77	1.34

28 days. The results in Figure 4 show that, even though the total deliveries are equivalent, the puff by puff deliveries differ markedly. For Cigarette T, 70% of the menthol on the tobacco, the menthol delivery increases in a fairly uniform fashion as the cigarette is smoked. For Cigarette F, 60% of the menthol on the filter, menthol deliveries are low in the early puffs and extremely high in the last puffs. This finding is in essential agreement with that given by Curran (1).

#### C. Mechanisms of Elution

Curran and Miller (2) discussed two factors, aerosol concentration and filter temperature, which affect the elution of semi-volatile materials from cigarette filters by smoke. Increases in either factor increase elution. It is probable that two other factors also contribute. The concentration of menthol in the TPM deposited on the filter will affect the vapor pressure of menthol above the filter fibers. The aerosol size distribution will affect the probability of capture of menthol molecules by aerosol particles. These four factors and their magnitudes for early and late puffs on the cigarettes are summarized in Table 2.

The increases in filter temperature, aerosol concentration, and aerosol unit surface area offer ready explanations for the large increases in menthol delivery found for the last puffs from the cigarette with a high concentration of menthol on the filter. What is surprising, however, is that no large increases in menthol delivery are seen in the other cigarette, even though 600 micrograms of menthol have been deposited on the filter by the 6th puff. The ratio of menthol/nicotine delivered for this cigarette, in fact, shows a slight decrease as the cigarette is smoked, Table 3. We conclude that the lowering of menthol vapor pressure by the TPM which is deposited on the filter accounts for the lack of any large increase in menthol transfer in the last puffs of this cigarette. The cigarette with most of the menthol on the filter has a higher concentration, over twice as great, of menthol in the deposited TPM. This is apparently sufficient to provide a high enough vapor pressure to give the large transfer found.

#### LITERATURE CITED

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